# 33 QUALITY ASSURANCE

## 33.1 Introduction

33.1.1 The purpose of this section is to provide a uniform Quality Assurance Program for the Toxicology Section of the Virginia Division of Forensic Science. In combination with the toxicology reporting guidelines, it is designed to establish a baseline or reference point of reliability and system performance.

33.1.2 It is expected that the analyst will report any unacceptable or anomalous behavior of any analytical system immediately to either their supervisor or the appropriate Instrument Specialist. It is further expected that appropriate actions will follow ASAP and be properly documented.

## 33.2 Reagents

33.2.1 Chemicals and solvents used in qualitative and quantitative analyses should be of at least ACS reagent grade or better.

33.2.2 All chemicals and commercial reagents are labeled when received with the date received and the initials of the individual receiving them, and again when opened. All chemicals and commercial reagents are replaced when their stated shelf life has expired or when they fail to perform adequately under controlled conditions.

33.2.3 All laboratory prepared reagents will be clearly labeled to reflect the reagent name, date prepared and examiner's initials. Any working bottles of reagent aliquoted from a stock bottle will be labeled in a similar manner. Reagent log sheets will specify the information that must be captured for each reagent. This will include a quality check to ensure that the reagent is functioning properly. A stock bottle of reagent may continue to be used until the aliquoted reagent fails the quality check. If this occurs, it is the analyst's responsibility to immediately dispose of the remaining reagent and to communicate this to all examiners with working bottles of the reagent. After the initial preparation, reagents are checked with each batch analysis through the use of calibrators and controls. The results of the QC check are recorded with the reagent logs or within the case file (notation that calibrators and controls were within acceptable QA/QC ranges is sufficient to document the reagent is acceptable).

33.2.4 Standards and reference material should normally be of USP-NF quality or purchased from a reputable manufacturer. Where practical, the identity and purity of reference materials should be verified (e.g. mass spectra, retention times or expected qualitative instrumental results).

## 33.3 Balances

33.3.1 All analytical balances will be checked quarterly for accuracy using Class S-1 weights or better. Record the weights in the balance log book with the date and analyst's initials.

33.3.2 The below listed balances are intended as examples of a balance class of type with appropriate check weights.

| Balance Type | Balance Examples | Check Weights |
| --- | --- | --- |
| Analytical | Mettler AE 160<br>Sartorius Basic | 10.0 (±0.2) mg<br>100.0 (± 0.2) mg<br>1000 (± 0.2) mg |

33.3.3 Accuracy and precision must be established after a balance has been put into service after purchase, calibration, maintenance or repair.

33.3.3.1 The check weights listed in the table in 33.3.2 are weighed and recorded on the balance logsheet.

33.3.3.2 The accuracy of each weight should meet the criteria in 33.3.2

33.3.3.3 If the weights fail to meet the criteria, recalibrate the balance. Log the calibration on logsheeet and record both the pre and post calibration measurements on the logsheet.

33.3.4 All balances are cleaned, serviced and calibrated every 2 years by an outside vendor. Record the service call on the analytical balance QC sheet kept with each analytical balance.

**33.4 pH Meters**

33.4.1 The pH meter is calibrated prior to each use using a 2 point calibration method. Refer to the individual pH meter's instrument manual for these procedures.

33.4.1.1 The reference buffers chosen should bracket the expected pH value range of the solution, if possible.

33.4.1.2 The pH values must be within ± 0.1 units of the pH value stated on each individual reference buffer's labeling.

33.4.1.3 The electrode slope must be within the range 92-102%.

33.4.1.4 If the calibration values are within the accepted limits, the pH meter is ready to use for reagents.

33.4.1.5 If the calibration values are not within the accepted limits, rerun and/or troubleshoot as necessary.

33.4.2 Rinse the electrode with $dH_2O$ or the buffer or standard, as appropriate.

33.4.2.1 Inspect the electrode for scratches, cracks or salt crystal deposits prior to each use. Clean or replace the electrode in response to the discovery of these deficiencies, as appropriate.

33.4.2.2 Do not wipe the pH electrode glass bulb.

33.4.2.3 Short term storage is any period of time that is less than long term storage, which is defined as greater than one week.

33.4.2.3.1 For short term storage: store the electrode in a bottle containing pH storage solution.

33.4.2.3.2 For long term storage: fill the reference chamber with filling solution and cover the fill hole. Put a few drops of pH storage solution into an electrode storage bottle or electrode protective cap and cover the sensing element and reference junction OR cover the sensing surface with the protective cap and store dry.

33.4.2.3.3 Do not store the electrode in $dH_2O$.

33.4.3 References buffers are not automatically replaced after their stated expiration dates as long as their calibration values remain within ± 0.1 units of the stated pH.

33.4.3.1 Keep the buffer bottle tightly sealed and free of contamination.

33.4.3.2 Do not reuse an aliquot of buffer or return it to the original bottle.

33.4.4 For best results, use good laboratory practices. Refer to the pH meter instrument manuals for recommendations of good laboratory practices, correct applications, problem samples and trouble shooting.

**33.5 Pipettes**

33.5.1 Semi-annually, check and record the performance and calibration of fixed volume, variable volume and repeater pipettes by weighing volumes of water with an analytical balance.

33.5.2 For repeater and variable volume pipettes, ten measurements will be taken at 2 different volumes. For fixed volume pipettes, 5 measurements will be taken at the specified volume of the pipette. The pipette precision must be within ± 5% CV and inaccuracy must be within ± 5%. For small volume pipettes (less than 20 µL), pipettes must be within ± 10% CV and ± 10% inaccuracy.

33.5.3 Record the QC data in a Pipette QC log. If the pipette is out of calibration, the pipette will be sent to the manufacturer for repair. Repair and/or maintenance of pipettes will be recorded in a log maintained for pipettes.

**33.6 Refrigerators/Freezers**

33.6.1 Weekly, check and record the temperatures on all refrigerators and freezers. Some reagents are temperature sensitive and therefore refrigerators should be between 2-8º C and freezers should be below -10ºC.

**33.7 Gas Chromatographs**

33.7.1 Most toxicology procedures are performed in batch and therefore most maintenance procedures are performed prior to running a batch of samples

33.7.2 Prior to running a batch analysis:

33.7.2.1 Change all septa and record in the logbook, date and initial

33.7.2.1.1 Unless a Merlin Microseal is installed

33.7.2.1.2 Unless the inlet is connected to a transfer line

33.7.2.2 Run performance check mix (calibrator or specific instrument check mix). If necessary, perform the following as necessary:

33.7.2.2.1 Change the liner

33.7.2.2.2 Replace the gold seal (for those split/splitless injector ports containing them)

33.7.2.2.3 Replace the gap column (if used)

33.7.2.2.4 Treat the GC with Silyl-8 (excluding instruments with NPD or ECD)

33.7.2.2.4.1 Condition column after treatment (increase injection temperature by 20º C and set oven temperature 10º C above final temperature for 15 minutes

33.7.2.2.5 Record all changes in instrument logbook, date and initial

33.7.3 Weekly

33.7.3.1 Check gas supplies and replace as needed

33.7.4 Semi-annually

33.7.4.1 Delete all non-MSD data files and sample tables that are more than one month old

33.7.4.2 Run a defragmentation program on computer hard drive

33.7.4.3 Archive MSD data to CD-ROM

33.7.5 Annually

33.7.5.1 Replace GC column as needed and record in log book, date and initial

33.7.6 For GC's equipped with NPD

33.7.6.1 Replace ceramic bead as needed

33.7.6.2 Replace collector and ceramics as needed

33.7.6.3 Replace or clean jet as needed

33.7.7 For GC's equipped with FID

33.7.7.1 Clean FID as needed

33.7.8 For GC's equipped with ECD

33.7.8.1 Bake-out detector as needed

33.7.9 For GC's equipped with MSD

33.7.9.1 Prior to use, autotune. Autotune maximizes instrument sensitiviy over the mass range, using PFTBA masses 69, 219 and 502.

33.7.9.2 Autotune criteria

33.7.9.2.1 The mass assignments shown in the upper profile part of the display should be within ± 0.2 amu of 69, 219 and 502.

33.7.9.2.2 Inspect the mass peaks in the upper profile part of the display for good peak shape (no peak splitting and resolution between mass 502 and 503).

33.7.9.2.3 The peak widths (PW) of the three peaks should be 0.6 ± 0.1 amu.

33.7.9.2.4 The isotope (iso) ratio figures (indicating the relative abundances of the naturally occurring isotopes) should be close to the theoretical values of 1.08 for m/z 69, 4.32 for m/z 219 and 10.09 for m/z 502.

33.7.9.2.5 Air and water leaks (mass 28 and 18) should be minimum.

33.7.9.3 The instrument shall be maintained to manufacturer's specifications, using repair/replacement guidelines set forth by the manufacturer, or to maintain optimum operating conditions.